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(21) International Application Number: PCT/US93/05301 (22) International Filing Date: 2 June 1993 (02.06.93) (30) Priority data: 07/897,688 12 June 1992 (12.06.92) US (71) Applicant: THE PROCTER & GAMBLE COMPANY [US/US]; One Procter & Gamble Plaza, Cincinnati, OH 45202 (US). (72) Inventor: HORD, Lee, Alan ; 3880 Irwin Simpson Road, Mason, OH 45040 (US). (74) Agents: REED, T., David et al.; The Procter & Gamble Company, 5299 Spring Grove Avenue, Cincinnati, OH 45202 (US).		(81) Designated States: AU, BR, CA, FI, JP, NO, NZ, RU, Eu- ropean patent (AT, BE, CH, DE, DK, ES, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE). Published <i>With international search report.</i>
(54) Title: PSYLLIUM DRINK MIX COMPOSITIONS (57) Abstract Psyllium husk-containing drink mix compositions comprising the divalent cation salt of strong inorganic acids selected from the group consisting of magnesium sulfate, calcium sulfate, calcium chloride, zinc sulfate, zinc chloride and mixtures thereof.		

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PSYLLIUM DRINK MIX COMPOSITIONS

BACKGROUND OF THE INVENTION

The present invention relates to psyllium husk-containing drink
5 mix compositions comprising the divalent cation salt of strong inor-
ganic acids selected from the group consisting of magnesium sulfate,
calcium sulfate, calcium chloride, zinc sulfate, zinc chloride and
mixtures thereof. These salts provide the benefit of reducing the
gellation rate of the psyllium husk when dispersed in an aqueous
10 solution.

Products containing psyllium seed husk are known (for example,
Metamucil®, sold by The Procter & Gamble Company). Such products are
useful for the benefit of normalizing bowel function and laxation. In
addition, recent research has demonstrated the effectiveness of
15 psyllium seed husk fiber in reducing human serum cholesterol levels
and in controlling blood glucose levels in diabetics.

Psyllium seed husk contains natural mucilage. It forms a
gellatinous mass on contact with water, and it exhibits poor dispersi-
bility and mixability in water. Dispersibility and mixability of
20 psyllium husk in aqueous solutions have been shown to be improved by
utilizing higher levels of sugar, and by coating the husk with
materials such as maltodextrin.

Once dispersed in the aqueous solution, the psyllium husk begins
to gel with an accompanying increase in the viscosity of the drink
25 solution. Typically, the consumer of the psyllium husk suspension
drinks the liquid suspension in a relatively short period of time
(less than about two minutes) in order to avoid having to drink an
aesthetically unacceptable high viscosity liquid (i.e., the solution
is considered too thick to enjoy drinking or difficult to drink). By
30 reducing the psyllium husk particle size it is possible to eliminate
the gritty texture of the psyllium husk yet maintain efficacy.
However, the smaller the particle size of the psyllium husk, the more
the rapid gellation rate is a consumer noticeable concern.

It is possible in one way to control the rate of gellation by
35 using acids to reduce the pH of the drink mix solution. However,
typical acids can impart too strong a flavor (e.g., a sour or bitter
flavor, especially at higher levels which may be desired to maximize
the reduction in gellation rate) to the solution, and this must be

consistent with the flavor system being used. Obviously, such an acidic medium is not suitable for flavor systems which require neutral or basic conditions. Also, except in certain controlled circumstances, acids are not suited for use in unflavored systems.

5 For these reasons, there continues to be a need for psyllium husk drink mix compositions having reduced (slower) gellation rates and improved aesthetics. It has been discovered that by adding the salts according to the present invention to psyllium husk-containing drink mix compositions the aesthetics of the drink compositions are im-
10 proved. For example, for the larger particle size psyllium husk which is less readily suspended, the husk which settles to the bottom of the glass has improved aesthetics such as lower viscosity for the last portions of the drink. For the smaller particle size husk which is more readily suspended, the aesthetics are improved by the liquid
15 suspension having a reduced gellation rate. This discovery is especially useful, for example, for allowing the use of low levels of acid or no acid (to reduce or eliminate the acid characteristic of drink compositions) to permit a wider variety of flavor systems (including "unflavored" versions of psyllium husk-containing drink mix composi-
20 tions), and to further reduce the gellation rate for compositions containing higher levels of acid.

It is therefore an object of the present invention to provide improved psyllium husk drink mix compositions having reduced gellation rates in aqueous solution and improved aesthetics. It is also an
25 object to provide drink mix compositions which are unflavored or are not acid flavored systems.

These and other objects of the present invention will become readily apparent from the detailed description which follows.

All percentages and ratios used herein are by weight unless
30 otherwise specified. Screen mesh sizes used herein are based on U.S. standards.

SUMMARY OF THE INVENTION

The present invention relates to psyllium husk-containing drink mix compositions. Such compositions comprise: (a) from about 10% to
35 about 99% psyllium husk; (b) from about 0.1% to about 50% of a divalent cation salt of a strong inorganic acid selected from the group consisting of magnesium sulfate, calcium sulfate, calcium chloride, zinc sulfate, zinc chloride and mixtures thereof; and (c) from about

0% to about 90% carrier materials; and wherein further said composition is in a form mixable with a liquid to form a suspension of the psyllium husk.

DETAILED DESCRIPTION OF THE INVENTION

5 The drink mix compositions of the present invention are psyllium-containing compositions in any form suitable for mixing with a liquid to form a psyllium husk suspension for oral consumption. Preferred form is a dry powder in bulk or unit dose form which readily mixes and disperses in the liquid. The components of the compositions according
10 to the present invention, and representative amounts, are described in detail as follows.

Psyllium Husk:

The psyllium husk used in the present invention is from psyllium seeds, from plants of the Plantago genus. Various species such as
15 Plantago lanceolate, P. rugelii, and P. major are known. Commercial psyllium husk include the French (black; Plantago indica), Spanish (P. psyllium) and Indian (blonde; P. ovata). Indian (blonde) psyllium husk is preferred for use herein. Also preferred is psyllium husk which is at least about 85% pure, more preferably at least about 90%
20 pure, and most preferably at least about 95% pure.

The psyllium husk is obtained from the seed coat of the psyllium seeds. It is typical to remove the seed coat from the rest of the seed by, for example, slight mechanical pressure, and then to use only the seed coat. The seed coat is preferably removed and sanitized by
25 methods known in the art. Preferred is sanitized psyllium seed husk having substantially intact cell structure, the sanitization having been accomplished by methods such as ethylene oxide sanitization and superheated steam sanitization (as taught in U.S. Patent No. 4,911,889, issued March 27, 1990 to Leland et. al., the disclosures of
30 which are incorporated herein by reference in their entirety). It is also preferred that the psyllium husk herein has reduced particle size.

Preferred psyllium husk utilized in compositions of the present invention have a substantial amount of small particle size psyllium
35 husk such that the psyllium husk comprises psyllium husk particle sizes distributed such that more than about 90% is smaller than about 45 mesh. More preferably, more than about 80% is smaller than about 50 mesh, further preferred is more than about 80% is smaller than

about 60 mesh, and most preferably at least about 80% is smaller than about 80 mesh. Further preferred particle sizes are distributed as follows: less than about 25% larger than about 60 mesh, and at least about 40% smaller than about 80 mesh. More preferred are particle size distribution of: less than about 10% larger than about 60 mesh, at least about 40% within the range of from about 80 mesh to about 200 mesh, and less than about 50% smaller than about 200 mesh. Particle sizes and particle size distributions may be readily determined by one of ordinary skill in the art, for example by sieving using an Alpine Laboratory Air Jet Sieve, Type 200 LS (sold by Alpine American Corp., Natick Mass.).

The drink mix compositions preferably contain from about 10% to about 99%, more preferably from about 20% to about 90%, most preferably from about 25% to about 75%, of psyllium husk.

15 Salts:

The salts useful according to the present invention are divalent cation salts of strong inorganic acids selected from the group consisting of magnesium sulfate, calcium sulfate, calcium chloride, zinc sulfate, zinc chloride, and mixtures thereof. Most preferred are calcium chloride and, especially magnesium sulfate.

Preferably the level of the salt is sufficient to reduce the gellation rate of the psyllium husk relative to the compositions without the added salt. Determination of whether the level of salt present in the psyllium husk-containing composition is a level whereby the gellation rate of the psyllium husk in an aqueous solution is reduced is readily made by simple experimentation, e.g. by comparing the rate of viscosity increase for the psyllium husk in a composition containing the salt versus the composition containing the same components but not the salt. Methods and equipment for measuring gellation rates and viscosity of psyllium husk are known, and such measurements and determinations can easily be made by one skilled in the art. For example, the Brinkman Viscometer may be used.

Compositions of the present invention therefore may comprise from about 0.1% to about 50% salts, preferably from about 0.1% to about 20%, and more preferably from about 0.5% to about 5% by weight of the drink mix composition.

Optional Carrier Materials:

Optional carrier materials useful for the compositions of the

present invention must be safe for oral administration to humans, and may be chosen by one of ordinary skill in the art as appropriate for the drink mix form and use intended for the product. Psyllium-containing drink mix products, methods for making, and carrier materials
5 useful for these products, are described more fully, for example, in U.S. Patent 4,459,280, to Colliopoulos et al., issued July 10, 1984; U.S. Patent 4,548,806, to Colliopoulos et al., issued October 22, 1985; U.S. Patent 4,321,263, to Powell et al., issued March 23, 1982; and U.S. Patent 4,828,842, to Furst et al., issued May 9, 1989; all of
10 which are incorporated by reference herein in their entirety. The drink mix compositions of the present invention comprise from about 0% to about 90%, preferably from about 10% to about 80%, and more preferably from about 25% to about 75%, of carrier materials.

Most preferred are products of the present invention in dry
15 powder form suitable for mixing in a liquid to form a psyllium-containing drink. Preferred carrier materials for such powder forms are known and are also described in detail, for example, in U.S. Patents 4,459,280 and 4,548,806, incorporated hereinbefore by reference. Preferred are such powders (preferably sugar free) comprising
20 maltodextrin. Also especially preferred are powders comprising agglomerates of psyllium and/or coated psyllium, especially agglomerated with maltodextrin and/or sucrose.

Agglomerating materials preferred for use herein are therefore known. These agglomerating materials include those selected from the
25 group consisting of water dispersible hydrolyzed starch oligosaccharide, mono-saccharide, di-saccharide, polyglucose, polymaltose, and mixtures thereof. Compositions of the present invention preferably comprise from about 0.5% to about 20% of agglomerating material coating on said psyllium husk, preferably from about 1% to about 10%,
30 and more preferably from about 1% to about 5%.

Hydrolysis of starch may be accomplished by a reaction of either acid, enzymes (e.g., alpha-amylase, beta-amylase or amyloglucosidase), or a combination of the two either together or reacted in series. The hydrolysis will follow different pathway depending on whether acids or
35 enzymes are used. The result is a mixture of oligosaccharides which may be separated for their different properties. The resulting separated water dispersible (preferably soluble) hydrolyzed starch oligosaccharides are classified by their reducing sugar content, i.e., the

mono- or di-saccharides such as glucose or fructose. The percent reducing sugar content in the particular hydrolyzed starch oligosaccharide is measured on a weight/weight basis as the Dextrose Equivalent (or "D.E."). Hydrolyzed starch oligosaccharides with a D.E. of from 0 to 20 are called maltodextrins. The solid maltodextrins have low to moderate sweetness, low to moderate hygroscopicity, solubility in water and alcohol, and have reduced browning. Above a D.E. of about 20 the hydrolyzed starch oligosaccharides are called syrup solids. The syrup solids are soluble but have a more noticeable sweetness and are more hygroscopic. Above a D.E. of about 30, the syrup solids become less desirable for use herein. A preferred water dispersible hydrolyzed starch oligosaccharide therefore has a D.E. of from about 0 to about 30. A preferred maltodextrin has a D.E. of from about 5 to about 20, more preferably about 10 (i.e., a reducing sugar content ratio of 10% w/w of the oligosaccharide).

The mono-saccharides are those carbohydrates that in general are aldehyde-alcohols or ketone alcohols that are a hexose or pentose and have a sweet taste. They are readily soluble in water and form crystalline solids. Examples of the di-saccharides are those carbohydrates which yield two mono-saccharides on hydrolysis. Examples of di-saccharides are lactose, sucrose and maltose.

Preferred compositions of the present invention comprise as part or all of the optional carrier material an edible acid. The term "edible acids", as used herein, means any water soluble acid material having a pK_a of less than about 5, preferably within the range of from about 2 to about 5, and is safe for ingestion by humans. Examples of edible acids include, but are not limited to, citric acid, ascorbic acid, malic acid, succinic acid, tartaric acid, phosphoric acid, monopotassium phosphate, and mixtures thereof. Preferred are ascorbic acid, phosphoric acid, malic acid, and citric acid, with citric acid being most preferred.

The compositions of the present invention typically comprise from about 0.1% to about 25% edible acid, preferably from about 0.1% to about 10%, and more preferably from about 0.1% to about 5%. Also preferred are compositions containing less than about 2% edible acid, more preferably less than about 1% edible acid, and most preferably less than about 0.5% edible acid.

Preferred compositions of the present invention are those which

have some or all of the edible acid coated on the psyllium husk, and further preferably such that the psyllium husk is agglomerated. Preferred single layer coating of the psyllium husk is achieved by utilizing equipment (referred to herein as single pass fluidizing powder wetting apparatus) which operates preferably by dropping a dry blend psyllium-containing material through a highly turbulent annular zone formed by a cylindrical wall and a rotating shaft with variously pitched attached blades. An edible acid-containing solution is preferably sprayed into this zone to contact a dry psyllium-containing blend. The resulting coated, preferably agglomerated, psyllium husk is dropped to a fluid bed dryer where the added solvent is removed. An example of this equipment is the Bepex Turboflex Model No. TFX-4 (sold by Bepex Corporation; Minneapolis, Minnesota) with a six square foot bed vibrating fluid bed dryer (sold by Witte Corporation, Inc.; Washington, New Jersey).

The psyllium-containing blend preferably comprises from about 25% to about 100% of psyllium. Optional components for the psyllium-containing blend include, but are not limited to, edible acid, sweetening agents (preferably low calorie sweetening agents), coloring agents, agglomerating materials (especially maltodextrin), dietary fibers such as brans (e.g., wheat bran; oat bran; rice bran) and/or pharmaceutical agents (e.g., aspirin; non-steroidal antiinflammatories; sennosides). Some or all of the salt may also be included in the psyllium-containing blend. As noted hereinbefore, it is preferred that the psyllium-containing blend be dry, but it is possible to utilize suitable solvents (e.g., alcohols and/or water) if one is careful, especially if water is utilized, not to cause substantial hydration and swelling of the psyllium, since this is expected to adversely affect the rate at which psyllium husk can interact with water or other fluids.

The solution mixture preferably comprises one or more edible acids to be sprayed onto the psyllium-containing blend along with also preferably comprising some or all of the salt. This may be prepared by selecting a liquid (e.g., alcohol and/or water) as appropriate for the materials being coated onto the psyllium husk. However, it is preferred that water be utilized. Preferred is also spraying the solution mixture onto a dry psyllium-containing blend. Preferably, when a spraying technique is used, the solution mixture is an aqueous

solution comprising from about 1% to about 50% (preferably from about 10% to about 25%) of the edible, water soluble salt and also from about 0% to about 50% (preferably from about 1% to about 20%) of edible acid. It is also optionally possible to repeat the coating and drying steps, thereby building up a coating on the psyllium husk which comprises several thin layers of the materials. In addition, other optional materials may be present in the solution mixture, such as coloring agents, pharmaceutical agents, and mixtures thereof.

Other methods for preparing compositions according to the present invention include dry blending the ingredients and other means of multiple layer coating of the psyllium husk. The latter may be accomplished by using, for example, fluid bed agglomerating equipment such as the Fluid Air, Inc. Model 0300 Granulator-Dryer.

Further, it is to be recognized that while the preferred drink mix compositions of the present invention are unflavored, it is possible to include with such preferred compositions sweetening agents, preferred being low calorie sweetening agents including, but not limited to, aspartame, saccharin, cyclamate, acesulfame, and mixtures thereof. Further, it is possible to use the present compositions as reduced flavor or non-flavored base formulations to make flavored compositions by adding flavoring agents, especially when the flavoring system is such that it is not compatible (chemically or aesthetically) with an acidic composition.

Method of Treatment

The present invention also relates to a method for providing laxation and regulating bowel function for a human in need of such treatment. This method comprises administering to a human in need of such treatment a safe and effective amount of a psyllium-containing composition of the present invention. Ingestion of from about 2.5 grams to about 30 grams per day of the psyllium fiber in a composition according to the present invention is appropriate in most circumstances to produce laxation. However, this can vary with the size and condition of the patient, and such matters will, of course, be apparent to the attending physician. However, since the psyllium material is nontoxic, even higher ingestion levels can be used without undue side effects. A typical dose for laxation purposes involves administering from about 3 to about 15 grams of psyllium fiber in one dose.

The present invention further relates to methods for reducing

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serum cholesterol levels in humans. These methods comprise orally administering to a human in need of having a lowered blood cholesterol level a safe and effective amount of an aqueous liquid suspension of a psyllium-containing composition of the present invention. Ingestion of compositions of the present invention comprising amounts sufficient to administer from about 2.5 grams to about 30 grams per day of psyllium fiber, preferably from about 5 grams to about 15 grams, is appropriate in most circumstances. However, this can vary with the size and condition of the patient, and the patient's blood cholesterol level. Such matters will, of course, be apparent to the attending physician. However, since the psyllium material is nontoxic, even higher ingestion levels can be used without undue side effects, keeping in mind the materials herein have the hereinbefore noted laxative effect.

Treatment of the patient to reduce serum cholesterol levels comprises chronic ingestion in order to lower and maintain the lowered cholesterol levels. Daily ingestion is preferred, and a daily ingestion of from about 5 grams to about 15 grams of the psyllium fiber is most commonly used, with said ingestion preferably being at 2 or 3 regularly spaced intervals throughout the day. Again, depending on the patient's size and cholesterol level in the patient's blood, this can be varied.

The following examples further describe and demonstrate embodiments within the scope of the present invention. These examples are given solely for the purpose of illustration and are not to be construed as limitations of the present inventions as many variations thereof are possible without departing from the spirit and scope.

EXAMPLE 1

	<u>Components</u>	<u>Weight %</u>
30	Psyllium ¹⁾	56.73
	Maltrin ²⁾	40.72
	Magnesium Sulfate	2.20
	Citric Acid	0.35

1) Psyllium husk of particle size 100% through 80 mesh.

35 2) Maltodextrin

This psyllium drink mix composition according to the present invention is prepared by agglomerating by spraying a dry blend of the psyllium husk and maltrin with an aqueous solution of magnesium

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sulfate and citric acid in a single pass agglomerator (as described in detail in European Patent Publication No. 412,604, published February 13, 1991, the disclosures of which are incorporated herein by reference in their entirety) and subsequently dried in a fluidized bed dryer. Consumption of one teaspoon of this composition as a suspension in 8 ounces of water is effective for providing laxation for a patient in need of such benefit.

EXAMPLE 2

	<u>Components</u>	<u>Weight %</u>
10	Regular Flavor Metamucil®)	98%
	Magnesium Sulfate	2%

1) Contains large particle size psyllium husk and dextrose.

This psyllium drink mix composition according to the present invention is prepared by dry mixing the ingredients. One teaspoon of this composition mixed with 8 ounces of water provides a drink having improved aesthetics and is effective for providing laxation for a patient in need of such benefit.

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CLAIMS

1. A psyllium husk-containing drink mix composition comprising:
 - (a) from 10% to 99% psyllium husk;
 - (b) from 0.1% to 50% of a divalent cation salt of a strong inorganic acid selected from the group consisting of magnesium sulfate, calcium sulfate, calcium chloride, zinc sulfate, zinc chloride and mixtures thereof; and
 - (c) from 0% to 90% carrier material;and wherein further said composition is in a form mixable with a liquid to form a suspension of the psyllium husk.
2. The composition according to Claim 1 wherein the salt is selected from the group consisting of magnesium sulfate, calcium chloride, and mixtures thereof.
3. A psyllium husk-containing drink mix composition comprising:
 - (a) from 20% to 90% psyllium husk;
 - (b) from 0.1% to 20% magnesium sulfate;
 - (c) from 10% to 80% carrier material;and wherein further said composition is in a form mixable with a liquid to form a suspension of the psyllium husk.
4. The composition according to any of Claims 1-3 wherein the carrier material comprises from 0.1% to 25% edible acid by weight of the composition.
5. The composition according to any of Claims 1-4 wherein the edible acid is selected from the group consisting of citric acid, ascorbic acid, malic acid, succinic acid, tartaric acid, phosphoric acid, monopotassium phosphate, and mixtures thereof.
6. The composition according to any of Claims 1-5 wherein the carrier material comprises from 0.1% to 10% edible acid by weight of the composition.

7. The composition according to any of Claims 1-6 wherein the edible acid comprises less than 2% by weight of the composition.
- 5 8. The composition according to any of Claims 1-7 comprising maltodextrin.
9. The composition according to any of Claims 1-8 wherein the psyllium husk is coated.
- 10 10. The composition according to any of Claims 1-9 wherein the psyllium husk is coated with maltodextrin.

INTERNATIONAL SEARCH REPORT

PCT/US 93/05301

International Application No

I. CLASSIFICATION OF SUBJECT MATTER (If several classification symbols apply, indicate all) ⁶		
According to International Patent Classification (IPC) or to both National Classification and IPC		
Int.Cl. 5 A23L2/38; A23L1/308; A23L1/0526		
II. FIELDS SEARCHED		
Minimum Documentation Searched ⁷		
Classification System	Classification Symbols	
Int.Cl. 5	A23L	
Documentation Searched other than Minimum Documentation to the Extent that such Documents are Included in the Fields Searched ⁸		
III. DOCUMENTS CONSIDERED TO BE RELEVANT⁹		
Category ¹⁰	Citation of Document, ¹¹ with indication, where appropriate, of the relevant passages ¹²	Relevant to Claim No. ¹³
X	CA,A,2 030 448 (HAUSER-KUHRTS) 23 May 1991 see page 16, line 13 - line 17 see page 16, line 25 - line 29 see page 18, line 9-19 see page 20, line 5-12 see page 21, line 1-7	1,4-7
Y	see page 23, line 13; claims 1,1,7,9,11,12,13,18; example 3 ---	1-3
Y	EP,A,0 040 654 (VITAPHARM) 2 December 1981 see claims 1,9,10; example ---	1-3
A	US,A,4 824 672 (C.E.DAY ET AL.) 25 April 1989 see the whole document ---	1-10
	---	-/--
<p>¹⁰ Special categories of cited documents:</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document but published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p> <p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step</p> <p>"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.</p> <p>"&" document member of the same patent family</p>		
IV. CERTIFICATION		
Date of the Actual Completion of the International Search	Date of Mailing of this International Search Report	
28 JULY 1993	10.08.93	
International Searching Authority	Signature of Authorized Officer	
EUROPEAN PATENT OFFICE	VAN MOER A.M.J.	

III. DOCUMENTS CONSIDERED TO BE RELEVANT (CONTINUED FROM THE SECOND SHEET)		
Category *	Citation of Document, with indication, where appropriate, of the relevant passages	Relevant to Claim No.
A	EP,A,0 362 926 (PROCTER & GAMBLE) 11 April 1990 see claims; examples see page 3, line 41 - line 47 -----	1-10
A	GB,A,2 224 628 (PROCTER & GAMBLE) 16 May 1990 see claims; examples -----	1-7
X	EP,A,0 412 604 (PROCTER & GAMBLE) 13 February 1991 see claims -----	1,8-10

**ANNEX TO THE INTERNATIONAL SEARCH REPORT
ON INTERNATIONAL PATENT APPLICATION NO.**

US 9305301
SA 75571

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Patent document cited in search report	Publication date	Patent family member(s)	Publication date
CA-A-2030448	23-05-91	US-A- 5118510	02-06-92
EP-A-0040654	02-12-81	None	
US-A-4824672	25-04-89	US-A- 4883788	28-11-89
EP-A-0362926	11-04-90	AU-B- 636304	29-04-93
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		US-A- 5219570	15-06-93

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